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COLLECTION AND CHARACTERIZATION
OF THE
SURFACE FILM OF WINDROWS IN MONTEREY BAY

James Ronald Reed

United States Naval Postgraduate School



THESIS

COLLECTION AND CHARACTERIZATION
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by

James Ronald Reed

June 1969

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Collection and Characterization
of the
Surface Film of Windrows in Monterey Bay

by

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Lieutenant (junior grade), United States Navy
B.S., University of Washington, 1968

Submitted in partial fulfillment of the
requirements for the degree of

MASTER OF SCIENCE IN OCEANOGRAPHY

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1969

REED, J.

ABSTRACT

A surface film collector was constructed and used to collect the surface film of windrows. The surface film was shown to be a complex mixture of primarily unsaturated esters of variable biological origin. Infrared, visible, and ultraviolet spectra are included.

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I. INTRODUCTION

Windrows are regions on the ocean surface where an organic film has been compacted by convergence of the surface water. Although these slicks are very wide spread and doubtlessly affect many physical, chemical, and biological properties of the ocean, the chemistry and source of the organic film has received very little attention. This lack of attention has probably been due to the lack of satisfactory sampling methods and, since the film is only a few molecules thick, to the small quantity of organic material actually present on the ocean surface in any particular area.

Studies were conducted to determine the feasibility of using a surface-film collector, designed after that of HARVEY [1966], to investigate the chemistry and source of windrows. Qualitative information on the composition and source of the organic film was obtained. The feasibility of more detailed investigations using the surface-film collector, and the possibility of obtaining large enough samples of the organic material to conduct chemical analyses was proven.

II. DESCRIPTION OF SURFACE-FILM COLLECTOR

The apparatus used to collect the surface film of the ocean surface is similar to that designed by GEORGE W. HARVEY [1966]. The collector (henceforth called the "skimmer") takes advantage of the surface tension of the sea surface. The skimmer consists of a partially submerged, rotating, stainless steel cylinder against which is pressed, near the top of the downward moving surface, a teflon blade to continuously remove the liquid film adhering to the surface of the cylinder. To prevent the collection of water other than the surface film the skimmer is pushed at a speed equal to or greater than the rotation speed of the cylinder.

A stainless steel cylinder 22 inches long and 12.5 inches in diameter was used. The cylinder was machined to a very smooth surface. The cylinder is supported above two styrofoam pontoons by aluminum structural members so that only 1.5 inches of the radius of the cylinder is immersed in the water (see figures 3 and 5). The pontoons are each 60 inches long, 15 inches wide, and 5 inches thick. The overall width of the skimmer is 69.5 inches and therefore is very stable. The skimmer weighs about 120 pounds and is easily handled by two persons (see figures 1 and 2).

The cylinder is turned by a Minn-Kota Electric Fishing Motor, Model UWJR, which is water cooled. The motor is immersed between one pontoon and the cylinder and is connected by a chain drive to a 200 to 1 reduction gear which is in turn connected by a chain drive to the shaft of the cylinder (see figures 2 and 4). The fishing motor operates on either a six or twelve volt automobile battery with two speeds at each voltage. The cylinder can be made to rotate at the following four speeds: 2.8, 3.9,

4.7, and 7 RPM. The advantages of this motor are that it operates in seawater without special protection, has four speeds, runs for several hours on one battery charging, and is inexpensive.

We used a teflon blade to remove the water, rather than a rubber blade, because it can be pressed tightly against the cylinder without the friction being so great as to seriously retard the rotation of the drum. Also the teflon is rigid and can be machined to a sharp edge where it makes contact with the cylinder. The teflon blade is bolted by means of adjustable wing nuts to a combination aluminum blade and trough which is inclined downward away from the cylinder and to one side so that the liquid flows to an aluminum container at one end of the cylinder. The aluminum container is attached by one wing nut so that it may be quickly and easily removed to empty the collected water into sample bottles.

The skimmer is pushed ahead of a 13-foot Boston Whaler outboard boat by means of an aluminum handle similar to that of a lawnmower (see figure 5) with the addition of cross braces to provide additional rigidity and also a platform to lie on when removing the collecting container. The handle positions the center of the cylinder about four feet ahead of the bow of the boat. The handle is tied by means of a rope to a cleat at the bow of the boat. A line runs from each side of the skimmer to a cleat at the stern of the boat to prevent yawing. By means of this arrangement, the boat can be turned within its own length without adjustment. The only maneuverability problem is speed. When moving from one windrow to another, the skimmer must still be pushed slowly to prevent the

pontoons from diving. The skimmer can be handled in up to three-foot swell and choppy water, but the increased turbulence at the forward side of the cylinder makes it doubtful that a satisfactory surface film sample could be obtained. Under such conditions, spray from the bow of the boat can contaminate the sample, and parts of the sample may be sloshed out of the container.

III. HISTORICAL SECTION

Considerable work has been done on the physics of wind-slick generation in the ocean. Very little has been accomplished on the source and chemistry of the surface film itself.

LANGMUIR [1938] first examined the physics of windrow generation and concluded that in addition to turbulence, "a series of alternating right and left helical vortices in the water having horizontal axes parallel to the wind" gave rise to the windrow pattern. This circulation pattern is now known as Langmuir circulation. Some factors that may give rise to the Langmuir circulation which collects the surface film into rows are the following: gravitational instability in a shear flow [WOODCOCK 1941; CSANDY 1963] , Coriolis force [STERN 1960; ARONS et.al. 1961; FALLER 1962, 1963] , the damping of capillary waves by the slicks themselves [STOMMEL 1952; KRAUS 1967] , the reduction by the slick of the frictional resistance to the air flow [WELANDER 1963] , and the turbulence in the water [MCLEISH 1968] .

As far as the chemistry of the surface film itself is concerned, the first paper was that of DIETZ and LAFOND [1950] . They suggested that natural slicks were films of oil from marine organisms, especially diatoms. They came to this conclusion on the basis of the following experiment: Crushing a sample of kelp and of diatoms and applying a drop of each to talc-covered seawater surfaces, they observed that the talc-covered surface was pushed back strongly by the diatom oil whereas the kelp oil only did so weakly. On this basis they suggested that the

organic film in windrows was primarily from the diatoms and not the kelp. They also measured the surface tension of water around a slick, one meter below a slick, and in the slick with a tensiometer and found that the surface tension in the slick was slightly less than that of pure seawater. They did not say how they sampled the water of the slick nor the exact lowering of the surface tension.

GARRETT and BULTMAN [1963] conducted laboratory experiments on the damping of capillary waves by organic monolayers. They conducted experiments with oleic acid, oleyl alcohol, ovomucoid, cetyl alcohol, and triolein films and found that they all behaved similarly with respect to wave damping even though they differed in chemical structure.

SUTTCLIFFE, et.al. [1963] bubbled air through filtered seawater and found organic particles in the spray droplets which aggregated when left undisturbed. These particles had a high phosphorus content and exhibited a high degree of surface activity. On this basis, they suggested that wave-induced bubbles and surface circulation may be an important mechanism for the production of surface active organic particles in the ocean.

One of the greatest drawbacks to the direct analysis of the surface film in slicks has been that of obtaining a large uncontaminated sample. SEBBA [1962] suggested the use of rising air bubbles but this is not practicable at sea because of excessive mixing of the surface layer with the subsurface water HARVEY 1966¹. GARRETT [1965] tried to use monel-metal screens and SIEBURTH [1965] stainless steel screens, but both had

problems with obtaining large samples and with preventing subsurface contamination.

The most important and best work on the collection of the surface layer is that of HARVEY [1966] . He designed a surface-layer sampler (from which ours was designed) which was able to collect large quantities of a thin surface layer without contamination. He said he was able to collect a layer only 60 microns thick. Samples obtained with his skimmer indicated larger amounts of organic material in the surface layer than in the subsurface water. The organic materials found included living plankton, disintegrated organisms, surface active substances, chlorophyll, and carotenoid pigments. Chloroform extracts of the collected surface layer were taken and an infrared spectrum was run. He said that the spectrum indicated large concentrations of organic material; but he did not give any indication as to the types of organic compounds present nor their possible source.

Since the probable source of the surface film is the natural lipids of marine plants the following references are included although they do not pertain directly to the surface film.

According to BONNER [1950] , the principal saturated fatty acids of plants are acetic, butyric, caproic, caprylic, lauric, myristic, palmitic, stearic, arachidic, lignoceric, and cerotic acids. The principal unsaturated acids of plants are caproleic, lauroleic, myristoleic, palmitoleic, oleic, petroselinic, ricinoleic, erucic, selacholeic, tariric, linoleic, linolenic, elaeostearic, licanic, parinaric, hydrocarpic, and chaulmo-

ogric acids.

HOOD, et.al. [1960] studied the organic matter in seawater collected from 10, 500, and 3000 meters. The fatty acids, determined by gas chromatographic techniques, present were C_{12} , C_{14} , C_{16} , C_{18} acids of both the saturated and unsaturated types. Palmitic and palmitoleic acids predominated (15% and 25% respectively) in the surface samples. A decrease occurred in unsaturation and chain length with depth.

KLENK, et.al. [1963] determined the unsaturated fatty acids of the lipids of some freshwater and marine algae. In marine green algae, the lipid content was similar to higher plant oils with C_{18} unsaturated acids predominating. The green algae also contained considerable amounts of polyunsaturated C_{16} acids. However, in the red algae C_{20} and C_{22} polyunsaturated acids predominated. The brown algae was intermediate between the green and red algae with some of the polyunsaturated C_{20} acids present as well as C_{18} polyunsaturated acids. In all the algae the C_{16} , C_{18} , C_{20} , C_{22} polyunsaturated acids found were of the linoleic and linolenic acid type.

KATES and VOLCANI [1966] determined the lipid components of marine diatoms. All the species studied contained glycerides, sulfoquinovosyl diglyceride, digalactosyl diglyceride, monogalactosyl diglyceride, phosphatidyl glycerol, lecithin, and phosphatidyl inositol as major lipid components. The majority fatty acid constituents were palmitoleic, palmitic, eicosapentaenoic, and eicosatetraenoic acids. Small amounts of hexadecadienoic, hexadecatrienoic, octadecadienoic, octadecatrienoic

and octadecatrienoic acids were present. The latter acid (linolenic acid), which is a major constituent in algae and higher plants, was only a minor constituent of diatoms lipids.

CHUECAS and RILEY [1966] determined the component fatty acids of some marine algae by means of gas-liquid chromatography. The principal saturated acids found were palmitic and myristic acids. The major unsaturated acids were octadecenoic and octadecadienoic acids (probably oleic and linoleic acids), but some species also contained up to 12% hexadecenoic acid. All the species contained significant amounts of eicosatetraenoic acid, and in some, eicosatrienoic and/or eicosapentenoic acids were present in small quantities. No C₂₂ unsaturated acids could be detected.

In summary the literature indicates that the lipid fraction of marine algae and diatoms consists primarily of esters of unsaturated fatty acids. These fatty acids contain 18 more carbons and are nonvolatile.

IV. DATA

Samples were collected on four occasions from windrows located approximately 500 to 1500 yards offshore from the Naval Postgraduate school beach located east of Monterey Marina. On all occasions, except the second, the sea was very calm with well-formed windrows. On the second occasion there was a choppy swell and the windrows were poorly defined except near the kelp beds. The skimmer performed satisfactorily on all four occasions. The removal of the organic film from an area swept by the skimmer could actually be observed by the formation of capillary waves where there had been none. Six to eight gallons of water were collected on each trip. The speed of the boat was about 2 feet per second and 3000 ml of sample was collected every 25 minutes. From this it was calculated that an average layer of .001 cm was removed from the sea surface. This thickness agrees well with that obtained by HARVEY [1966].

The samples were filtered to remove the particulate matter, and then the organic materials were removed by continuous extraction with ether for periods of 24 to 72 hours. The ether containing the organic compounds was then evaporated under vacuum. Although it had a very distinct odor, the amount of organic material from each extraction (2000 to 3000 ml of water sample) was very small; therefore all the extractions were combined into two samples. One sample contained those extractions which did not contain any visible chlorophyll and the other contained those that did. The non-chlorophyll sample ("greasy sample") was a brownish grease and the chlorophyll sample ("green sample") was a

bright green grease. Each of the extracts still appeared to be less than a gram, but this was expected since the film on the ocean surface is at most only a few molecules thick.

Since there are many kelp beds in the vicinity of the windrows in Monterey Bay, the kelp was postulated as a possible source of surface-active organic material. A sample of kelp was therefore ground-up in a blender without making an effort to break the cell walls. The ground-up kelp material was then extracted with ether, and the ether evaporated. A green organic material resulted that was somewhat similar to the green windrow extract except for the fact that it had a different odor.

Each of the three extracts was then dissolved in 1.5 ml of chloroform in order to run infrared, ultraviolet, and visible spectra and to obtain separations by chromatography.

The infrared spectra was taken on a PERKIN-ELMER 337 Grating Infrared Spectrophotometer. The infrared spectra obtained are shown in figures 6, 7, 8, 9, 10, 11.

Since the visible and ultraviolet spectrophotometer is much more sensitive than the infrared spectrophotometer, the 1.5 ml chloroform solutions had to be diluted by 100:1 or 1000:1. The visible and UV spectra were taken on a BECKMAN-DB Spectrophotometer. These spectra are shown in figures 12, 13, and 14.

Separation of the components of the extracts was attempted by gas chromatography using a HI-FI 600C Gas Chromatograph and a 5% carbowax 20M on chromosorb T column. Column temperatures of up to 250° C

were used. No components of the mixtures came off the column even after two hours.

Separation of the components of the three samples was accomplished by means of thin-layer chromatography. Two different solvents were used: 90% chloroform-10% carbon tetrachloride solution and 80% benzene-20% heptane solution. Representations of the separations obtained are shown in figures 15 and 16.

V. INTERPRETATION

The data suggests that the surface film is composed primarily of a mixture of unsaturated esters from a variety of biological sources .

The infrared spectra of the three extracts are all similar. They are typical unsaturated ester spectra with a strong carbonyl peak at about 1720 cm^{-1} and a C=C peak at about 1590 cm^{-1} . The $-\text{CH}_2-$ peak is less strong than the carbonyl peak. This suggests that the $-\text{CH}_2-$ peak is probably mainly due to the carbon chain of the esters rather than to hydrocarbons . Hydrocarbons are therefore probably present in the surface film only in small amounts .

The visible spectra of the green windrow extract and the kelp extract indicate the presence of chlorophyll a and b and carotene beta pigment. The visible spectrum of the greasy extract does not give any information.

The ultraviolet spectra of all three extracts indicate conjugated unsaturation since there is strong absorption at about 240 millimicrons .

The fact that no components of the mixtures came off the gas chromatographic column indicates that the esters are of high molecular weight and non-volatile . This non-volatility is expected if the organic film is to remain stable on the ocean surface . If a different gas chromatograph or column were used or if the esters were broken down into their simpler derivatives , it would probably be possible to separate the components of the mixtures .

The thin-layer chromatographic plates indicate that the three extracts are different mixtures. In the $\text{CHCl}_3\text{-CCl}_4$ solution, the kelp extract gives two spots, the greasy extract three, and the green extract two, including the original spots which remained visible. In the benzene-heptane solution, the kelp extract gave one spot, the greasy extract two, and the green extract two, including the original spots. All the spots have different R_f values and colors. This indicates that the slick-forming material is of variable composition and does not entirely originate from the kelp.

In summary, it can be stated that the slick-forming material is a complex mixture of non-volatile, high molecular weight esters of variable biological origin. The esters are primarily unsaturated. On occasion, chlorophyll and carotene pigments are present in the film. It can be surmised that these esters are the same or similar to the high molecular weight esters reported in the literature as being the components of the lipid fraction of marine algae and diatoms.

VI. SUGGESTIONS FOR FURTHER WORK

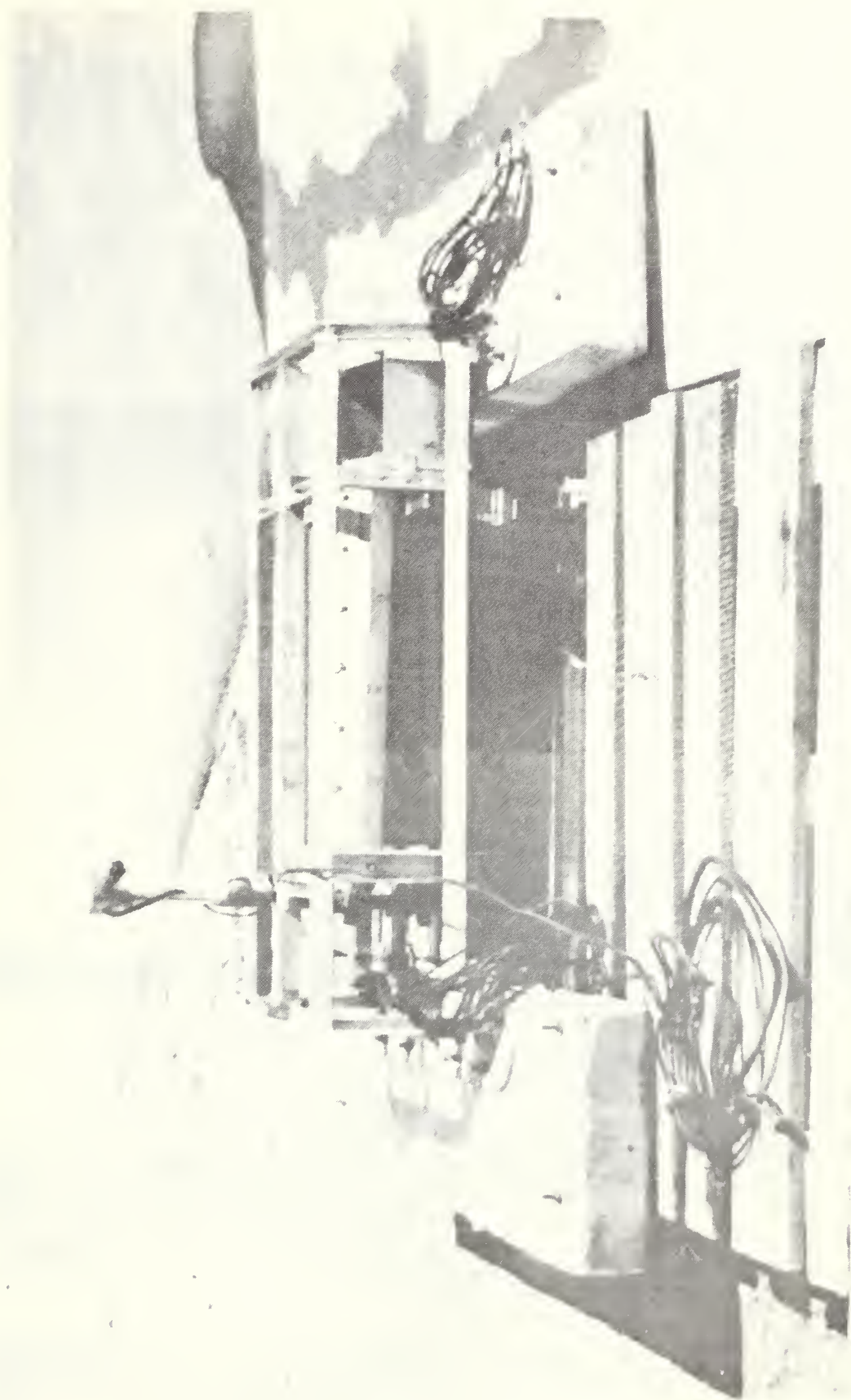
Qualitative information on the chemistry and source of slick-forming material was obtained, and the feasibility of using the skimmer for a more detailed study of the chemistry of windrows was proven. Our suggestions for future investigations fall into three categories: improvements of the skimmer, oceanographic investigation, and chemical analyses of the organic film.

The skimmer should be made larger so that studies can be conducted in larger waves, and water collected more rapidly. Even at the present size, higher bows should be built onto the pontoons to allow higher speeds when moving from one windrow to another. There was a tendency for the teflon blade to warp causing a loss of part of the film adhering to the cylinder. A tension system should be designed to prevent this. Some type of cover should be built over the collecting trough and container to prevent contamination of the sample from spray. A method of rapidly clamping the handle of the skimmer to the bow of the boat should also be devised. In order to eliminate lying on the handle to remove the collecting container, a system that syphons or pumps the sample back to the boat should be designed. The importance of the cleanliness of the surface of the cylinder cannot be overemphasized. Small amounts of dirt or corrosion on it tend to break the film adhering to the cylinder and cause it to be lost.

Windrow samples should be obtained farther out to sea and in a variety of locations under varying oceanographic conditions. Seasonal

and diurnal variations in the composition of the surface film and variations with such environmental parameters as temperature, wind velocity, current velocity, light intensity, and wave height should be observed. Correlations between the composition and number of windrows with biological activity should also be found. Measurements of heat and light transmission and gas diffusion through the film and the surface tension of the film should be made. The lipid composition of phytoplankton, zooplankton, marine benthic algae, and fish oils can be compared with the composition of the film to determine its primary sources.

Several large extractors should be constructed so that large quantities of water can be processed to obtain large enough samples to conduct a variety of chemical analyses and separations. Also a means of evaporating large quantities of ether rapidly without losing the ether would be beneficial. The following methods of analysis of lipids can be attempted if enough sample is available: conversion of the esters to their free fatty acids, determination of the saponification number, determination of the Reichert-Meissel number of any volatile fatty acids present, separation of the saturated and unsaturated fatty acids or esters, determination of the Iodine number, the separation of the esters by gas chromatography or if this is not possible then the simpler derivatives of the esters can be separated, and the determination of the NMR spectra of the various esters present.



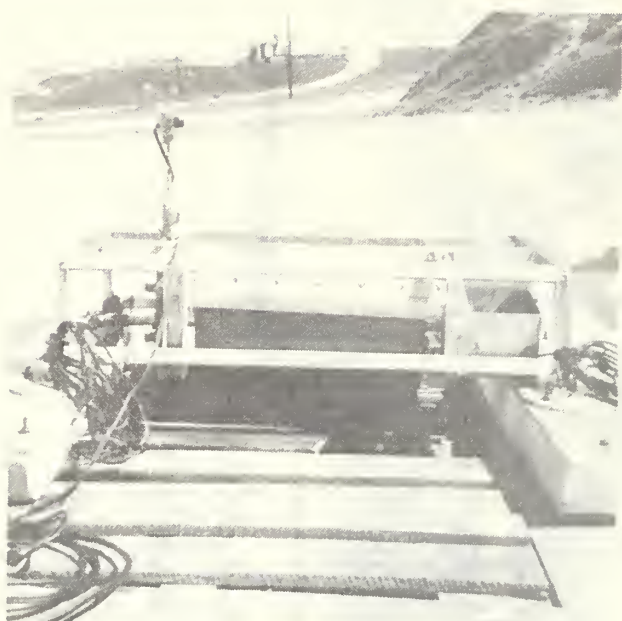


Figure 2

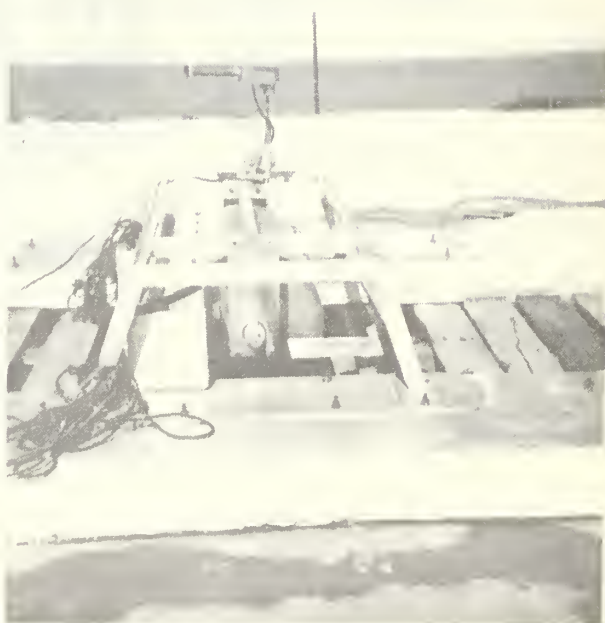


Figure 3

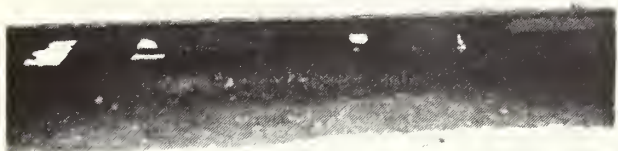
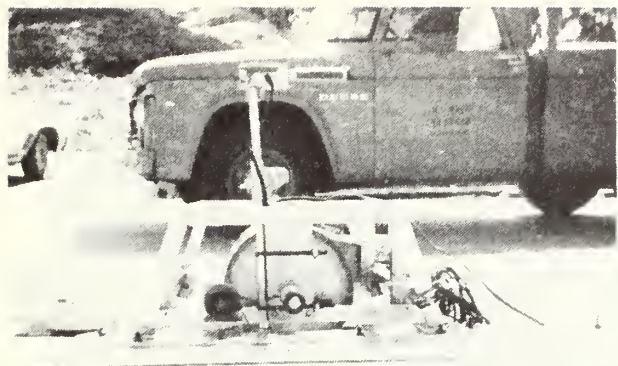
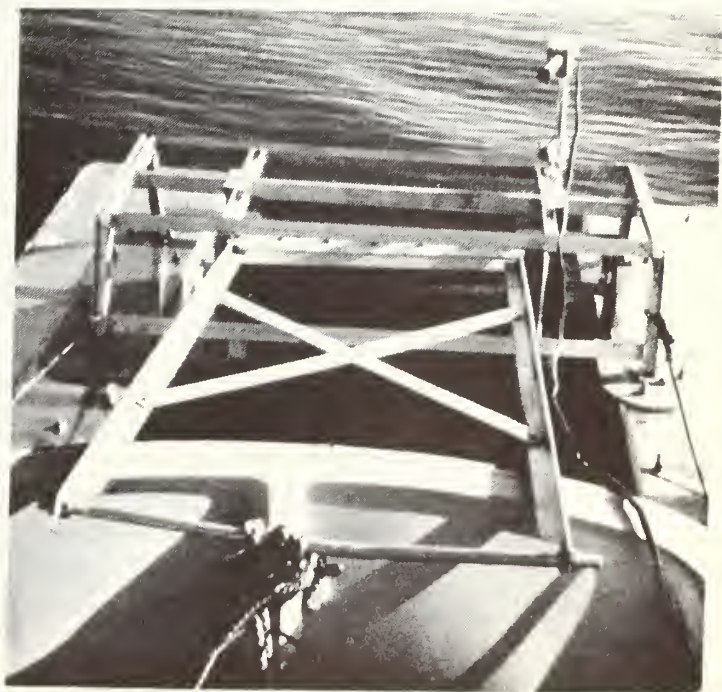


Figure 4

Figure 5



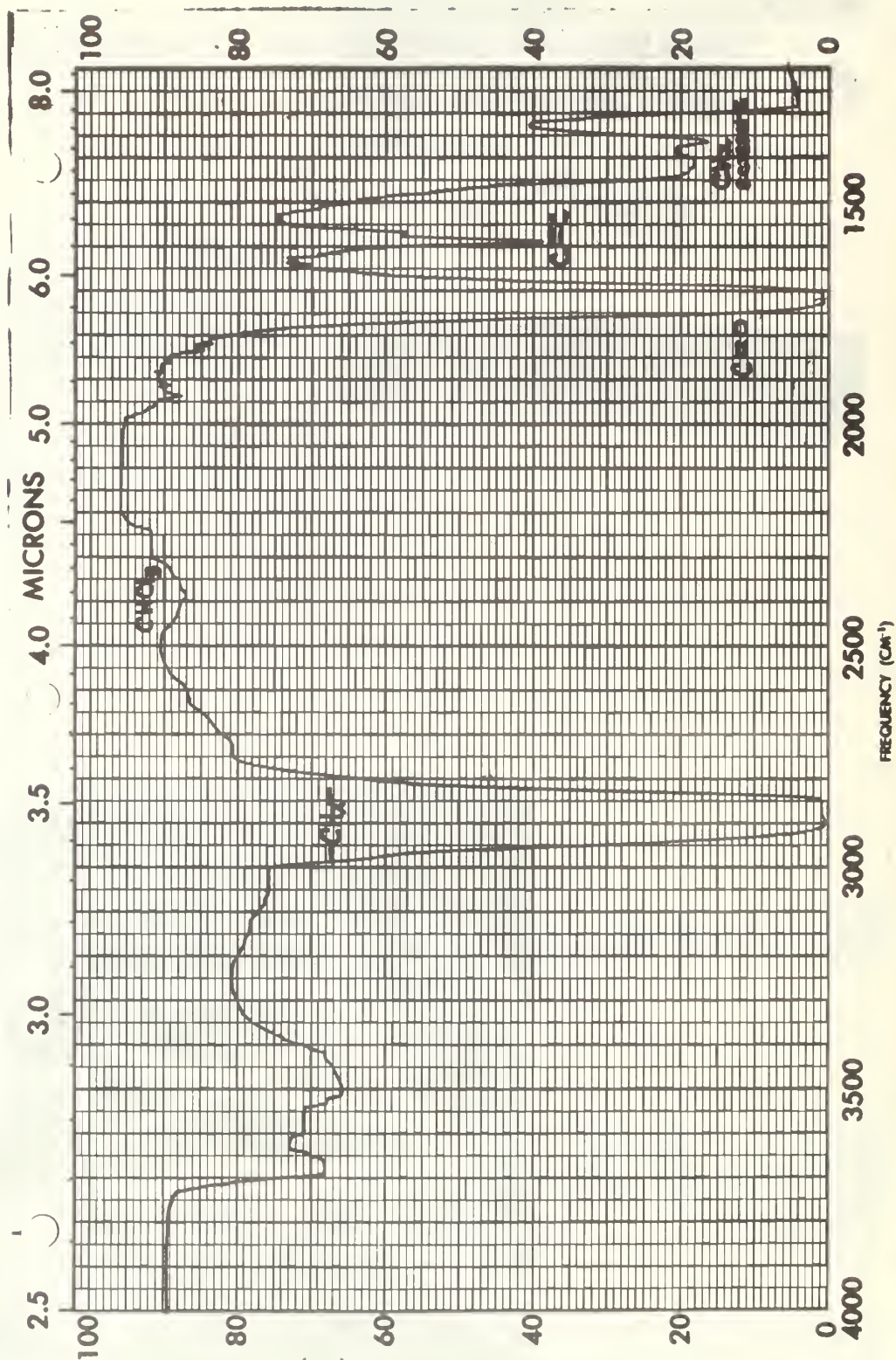


Figure 6
IR Spectra of Greasy Windrow Extract

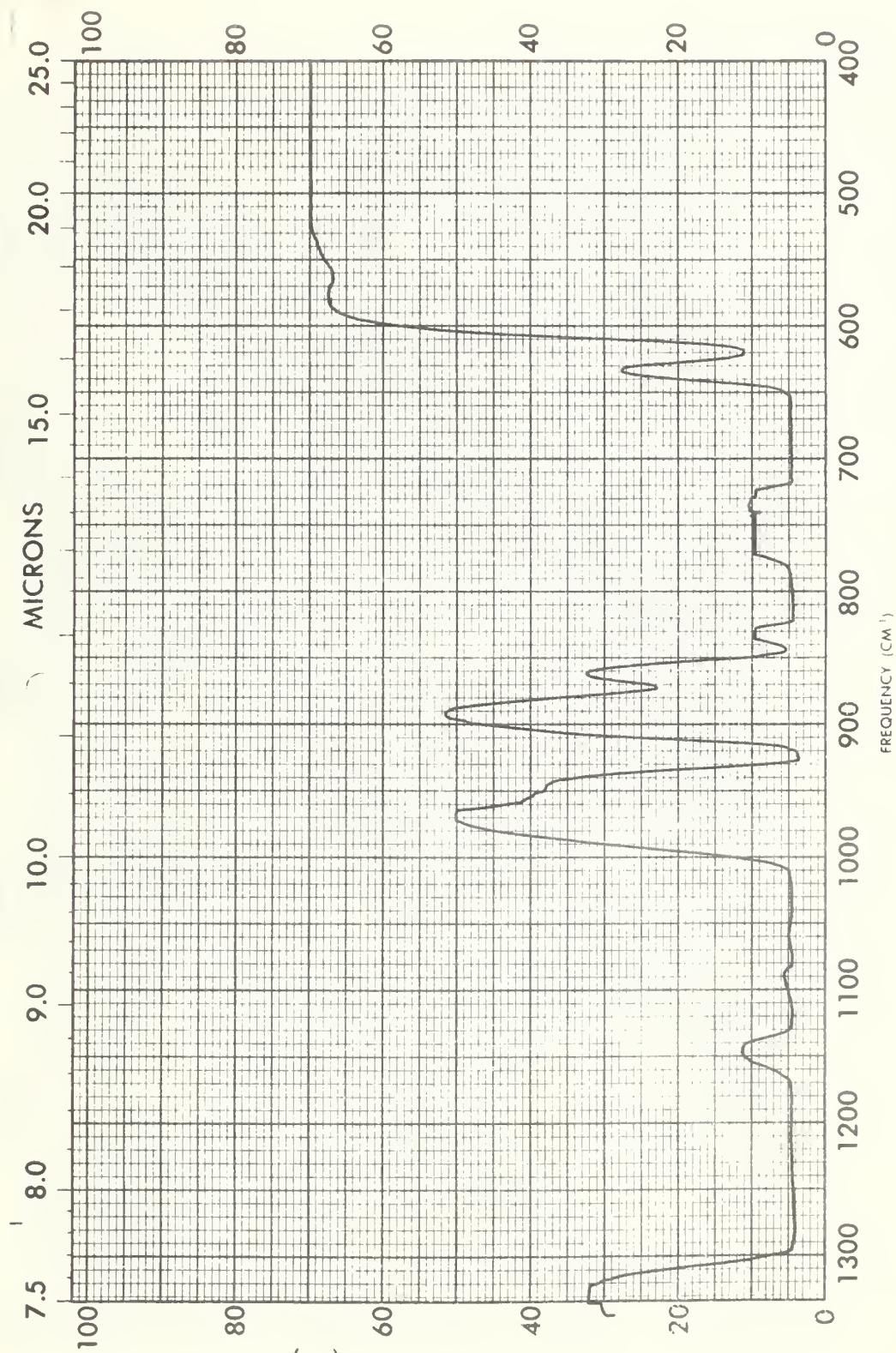


Figure 7
IR Spectra of Greasy Windrow Extract

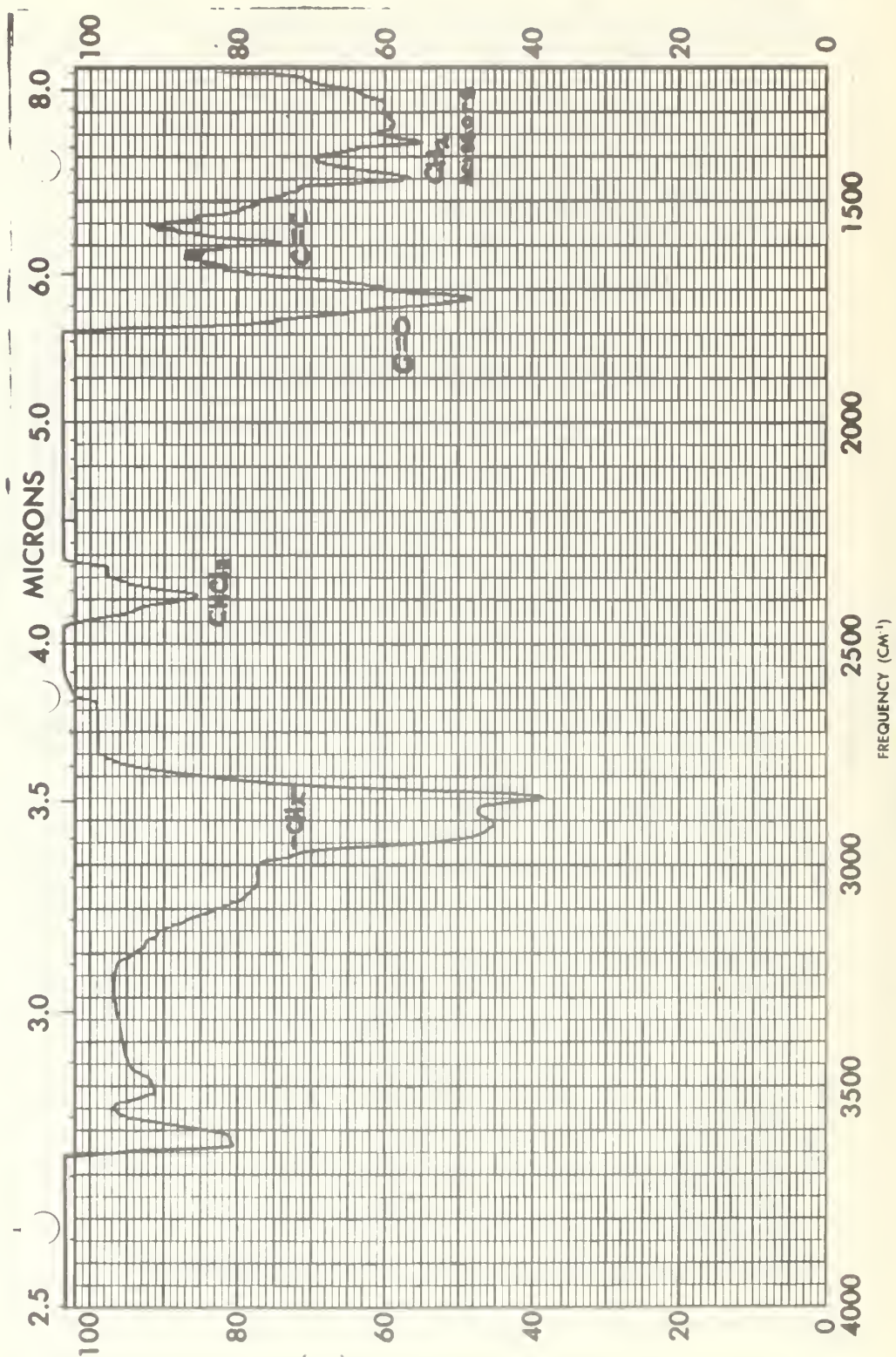


Figure 8
IR Spectra of Green Windrow Extract

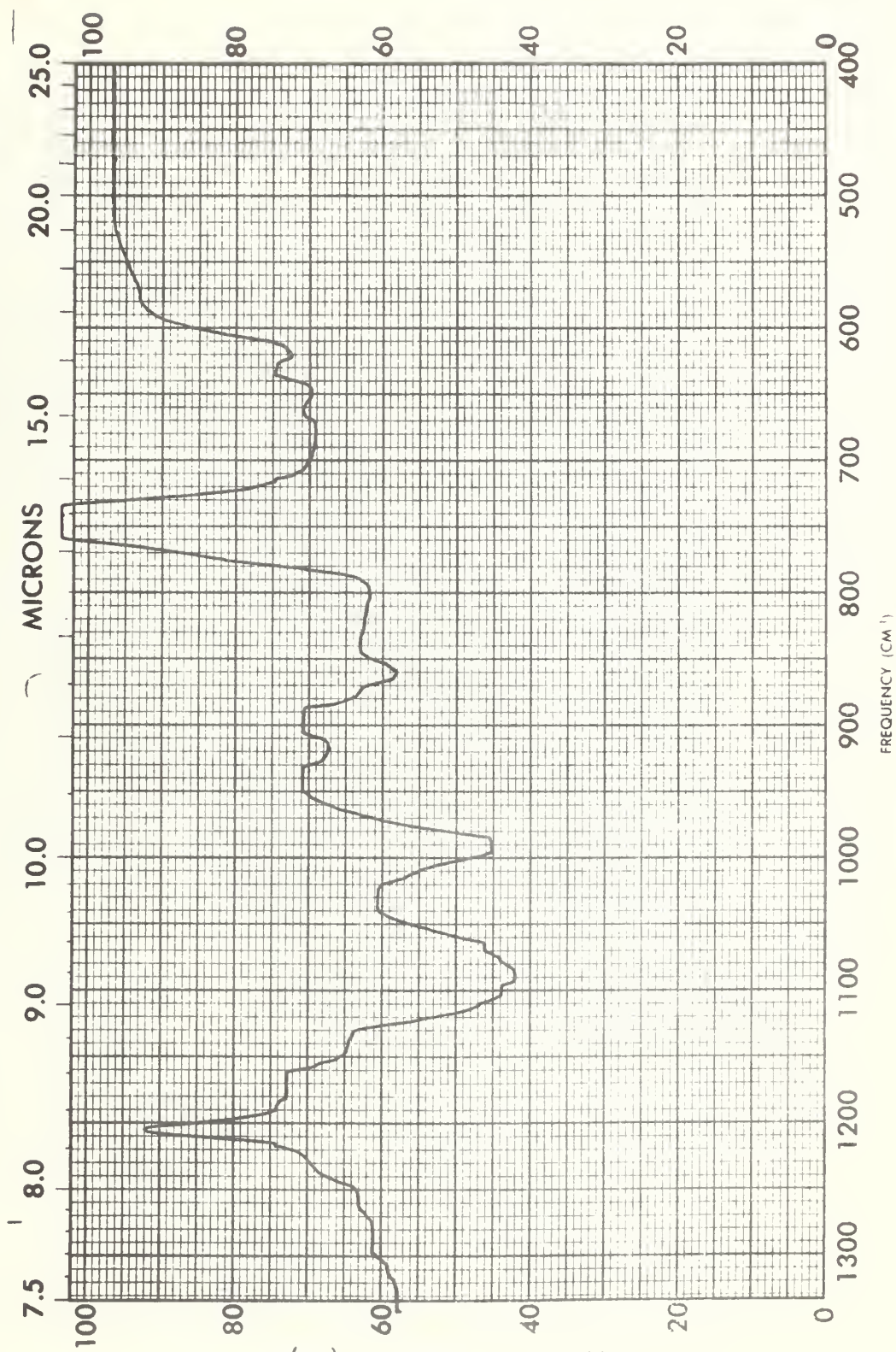


Figure 9
IR Spectra of Green Windrow Extract

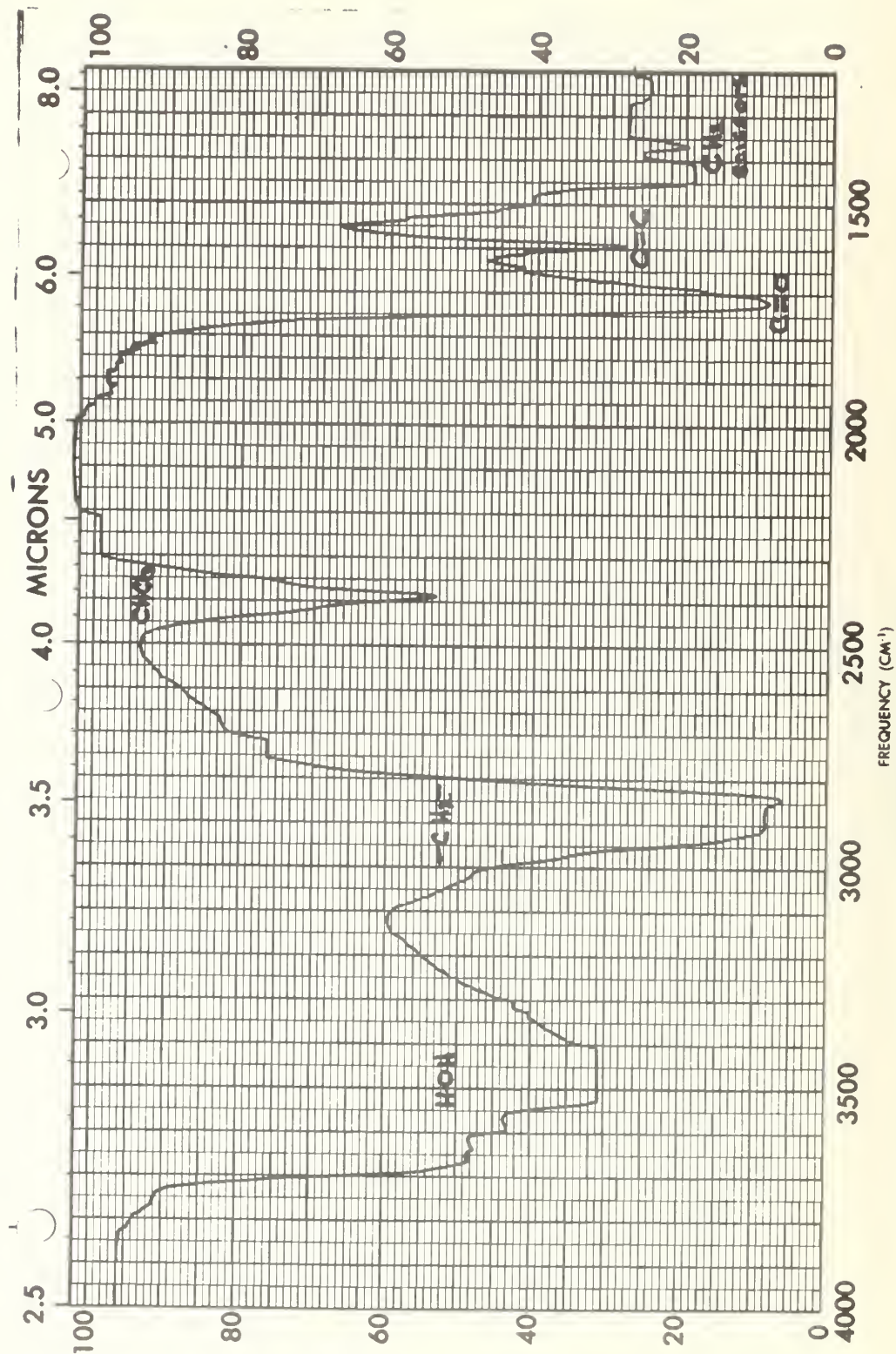


Figure 10
IR Spectra of Kelp Extract

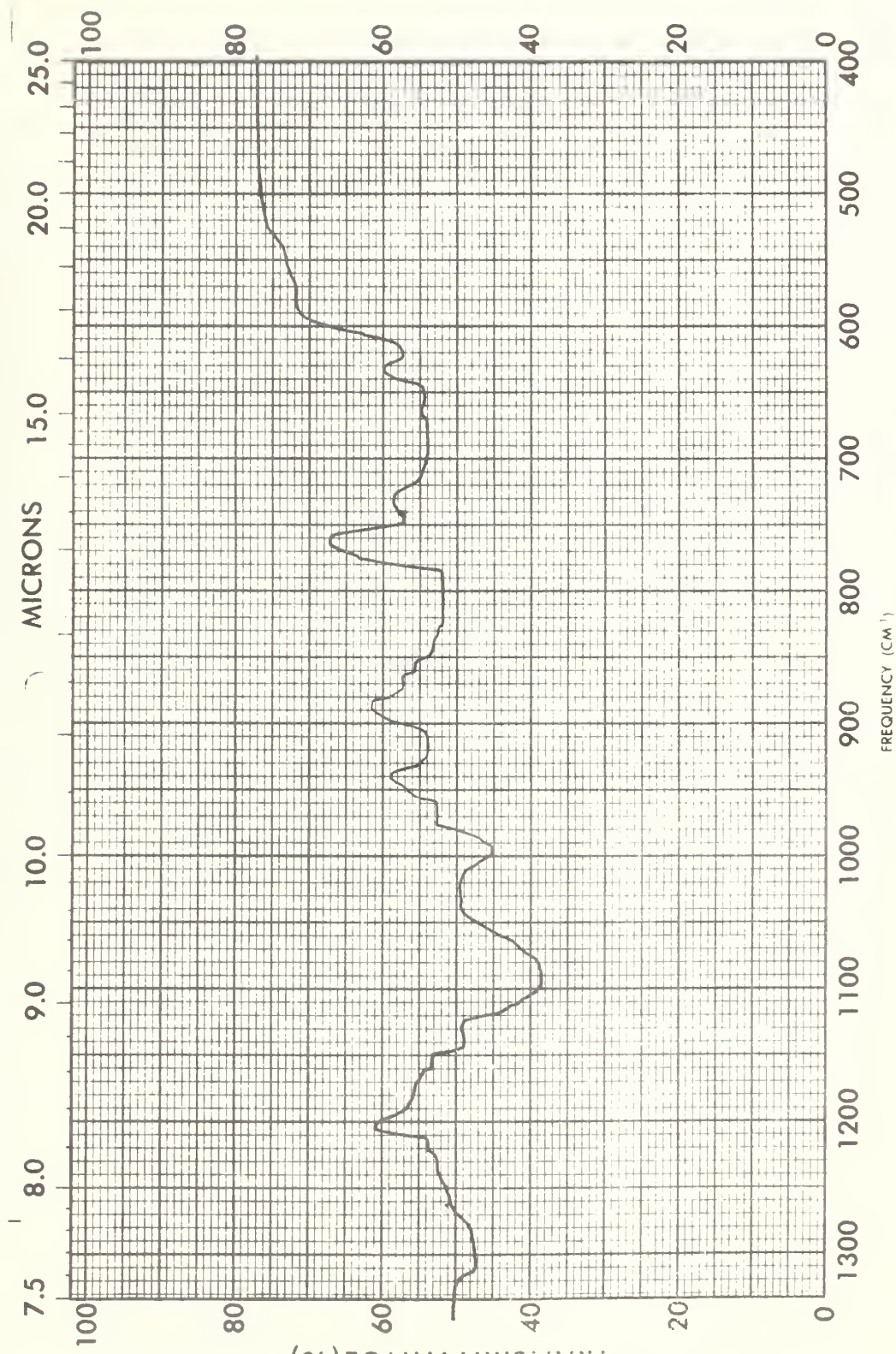


Figure 11
IR Spectra of Kelp Extract

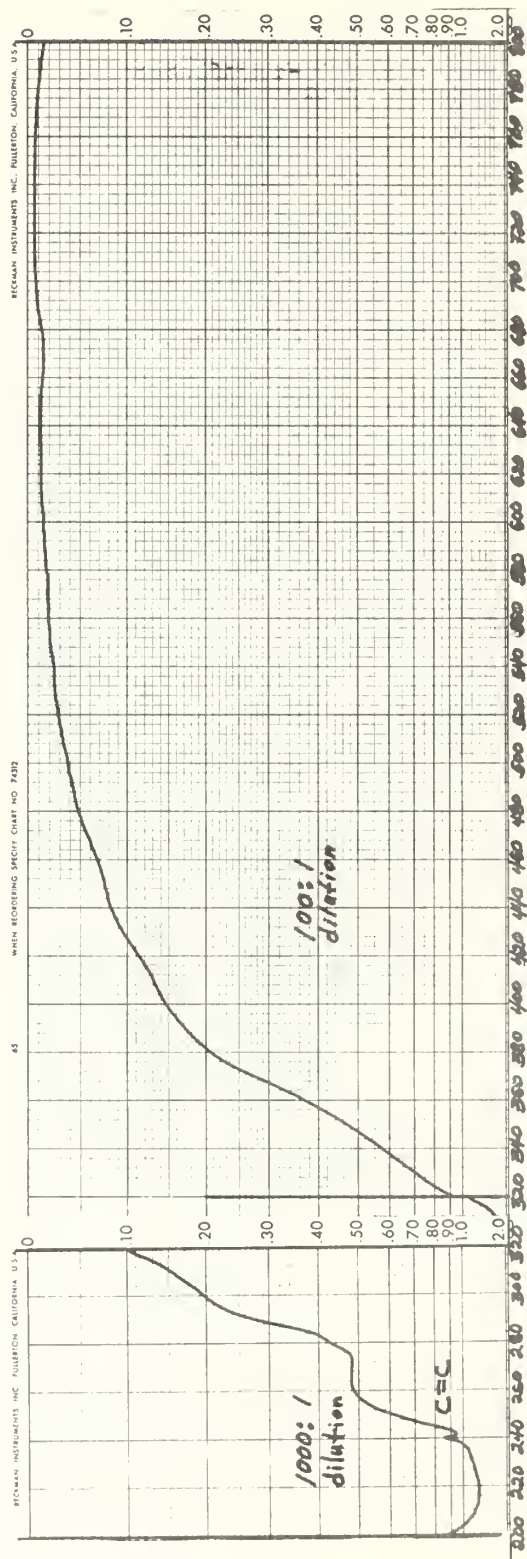


Figure 12
Visible and UV Spectra of Greasy Windrow Extract

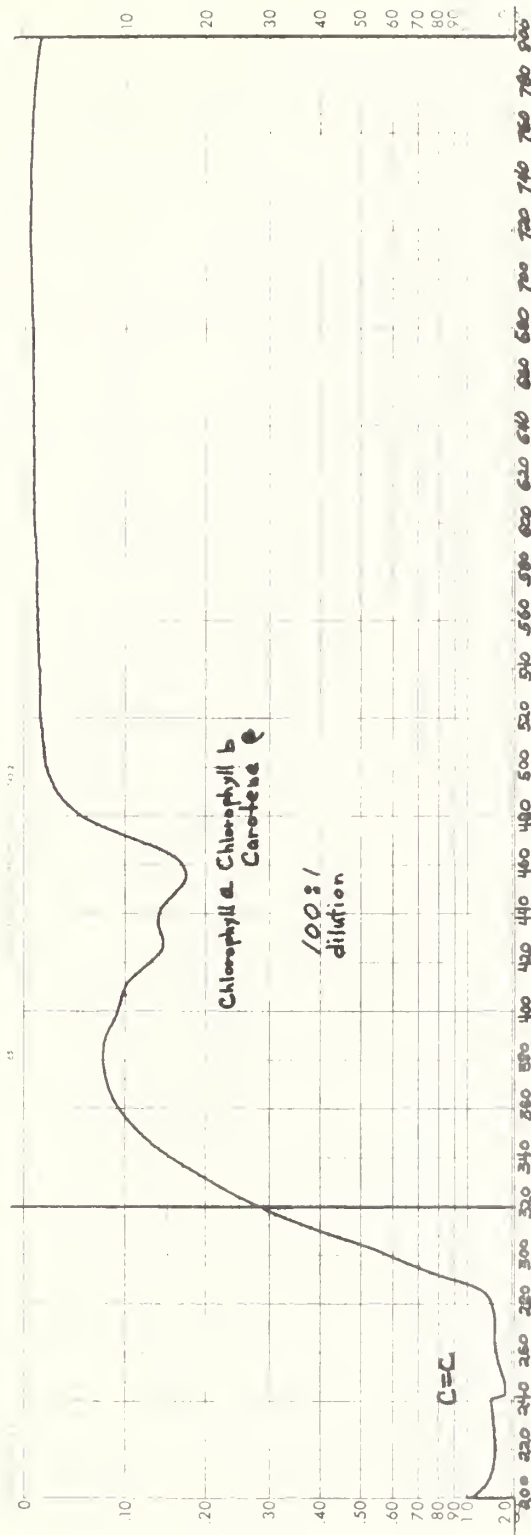


Figure 13
Visible and UV Spectra of Green Windrow Extract

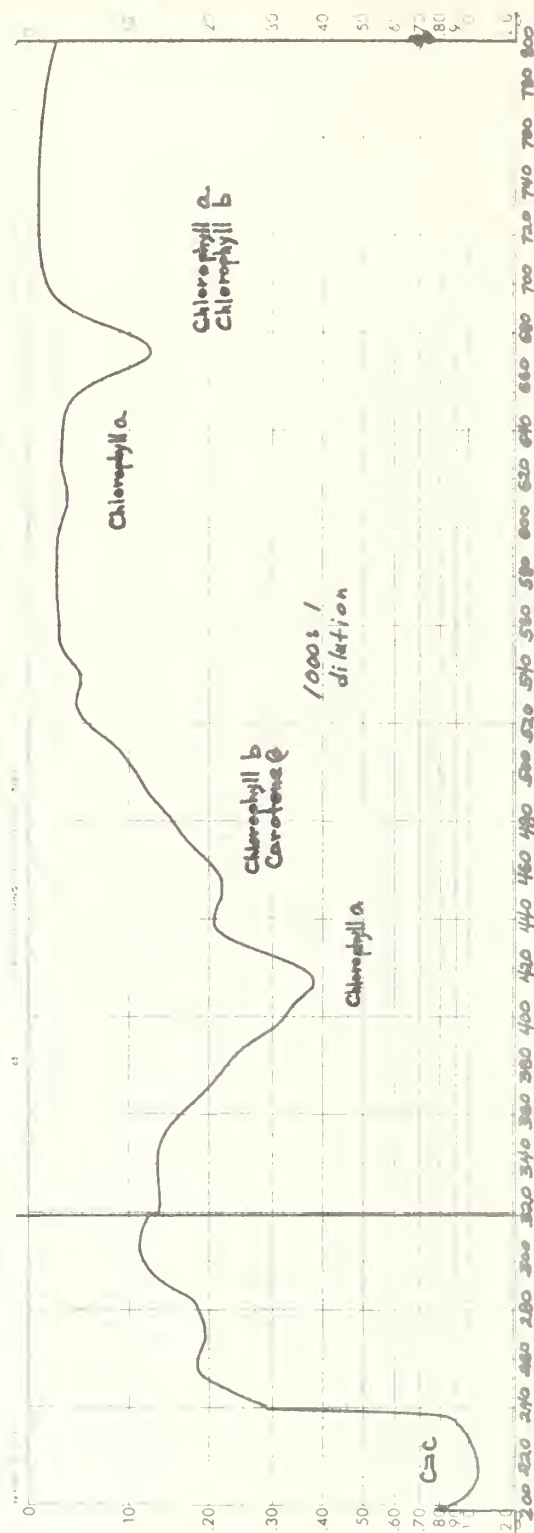


Figure 14
Visible and UV Spectra of KelpExtract

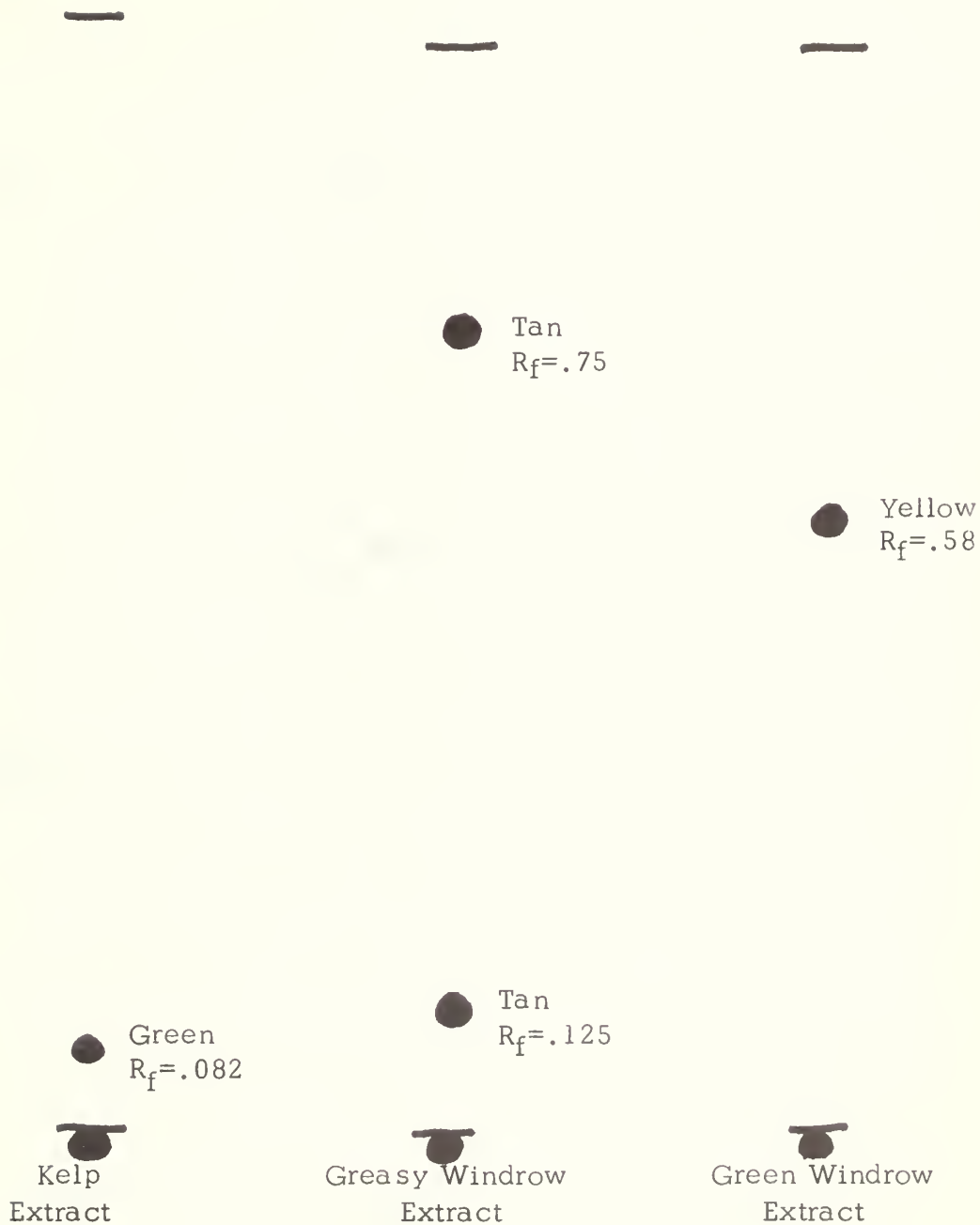


Figure 15
Thin-Layer Chromatography
90% CHCl_3 -10% CCl_4

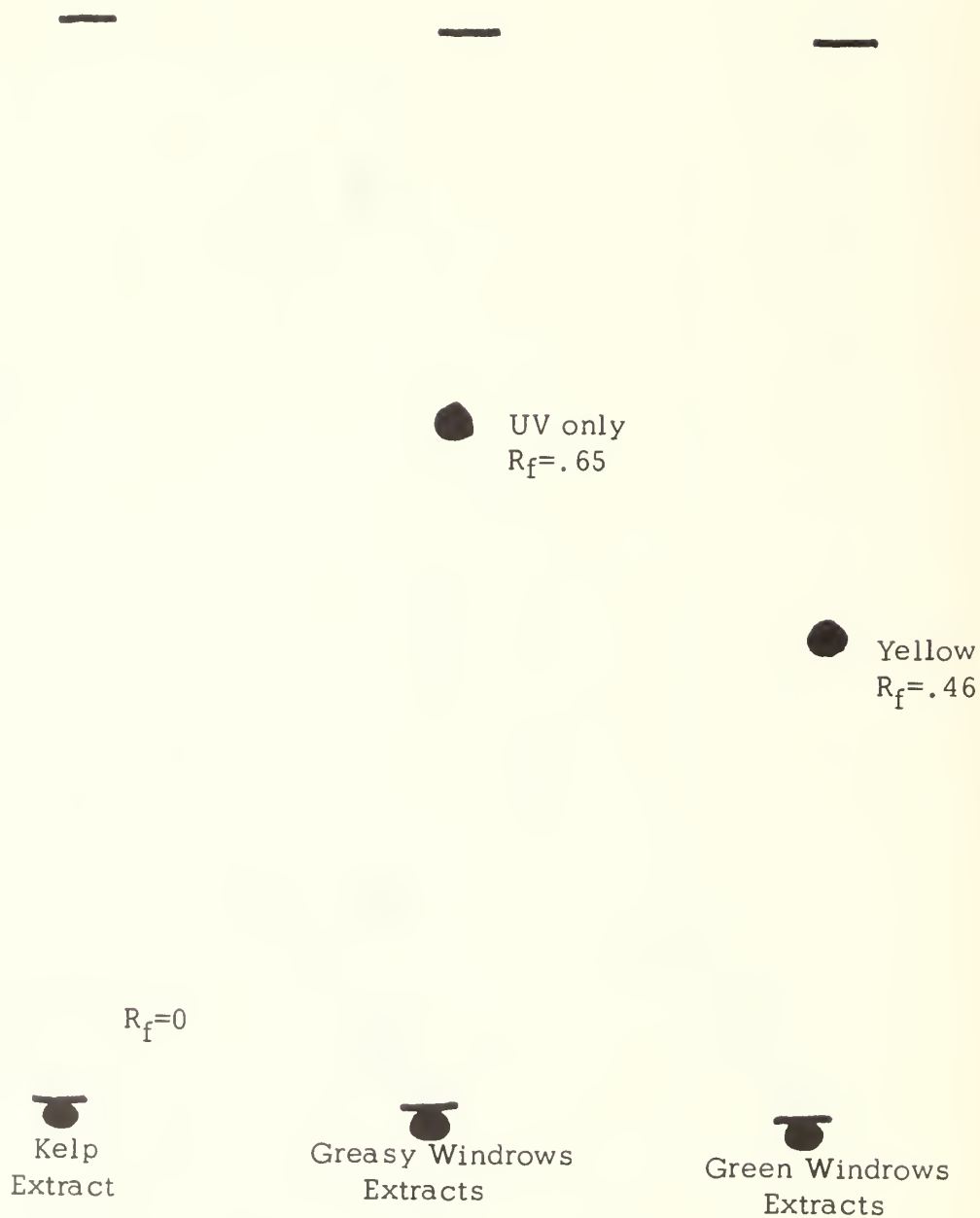


Figure 16
Thin-Layer Chromatography
80% Benzene-20% Heptane

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